EP at DESY- study optimum parameters

You need infrastructure to study!! (Statistic)

It influences possible studies and results

Base line: How to start for building a new facility

Apparatus

- Design (mechanic)
- Administration
- Sensors
- Acid
- Space

Process Parameters

- Voltage /Current
- Temperature
- Field-flatness

Design (mechanic)

DESY EP designed for

- Mono cells and cavities up to 2*9 cell super-structure
- Designed according German laws applying for Hamburg
- Available space
  - No basement allowed (water pipes and drains below EP space)
  - (➔sump + 2nd pump)
  - Height limited annex to building (clearance 1 inch on top)
- EP apparatus is an extension to the existing infrastructure
  - =>Limited space for acid storage
  - =>Limited quantity of rinsing water
  - =>Use of existing neutralization
  - =>location and connection to UPW and neutralization of rinsing water

German laws applied for

Explosion safety ➔ cabeling + sensors + material

Need to

- Reduce explosion risk on the point of appearance (Cavity)
  - ➔ N2 Overlay and H2 sensor on point of appearance
  - (+ 70000$ for explosion safe electric)
- Cavity has to be H2 leak tight
- Rotating shaft seal and gaskets to be controlled
- H2 / O2 not expected in drain acid H2 leaks on
- System (Cavity) at normal pressure

Acid

- DESY EP is annex to the existing facility
  - (reduced administration for license)
  - Acid volume limited to a total of 250 Kg
  - ➔ single barrel ; No mixing station!
  - ➔ External heat exchangers
  - ➔turn around industrial barrels
  - ➔ Safety volume (Housing has to hold 200 l acid)
  - (Nomura plating 1t in basement container)

Ventilation

- Exchange room volume 10 / hour➔ Size of housing + Gas scrubber

HF gases => no mixing allowed

➔total amount to be measured in 1200 l/h exhausting volume

Exhausting pipes located inside the work space area

➔ Max HF concentration 1,7 ppm ==Stop process

➔ Max HF concentration >3 ppm stop apparatus

Safety

- Definition of Sensors; Safety features
- personnel and infrastructure , containments

Rinsing water

- DESY neutralization stand allows 200l per hour of Ph 1

➔ Reality 200 l of Ph 0 –0,2 from first rinse

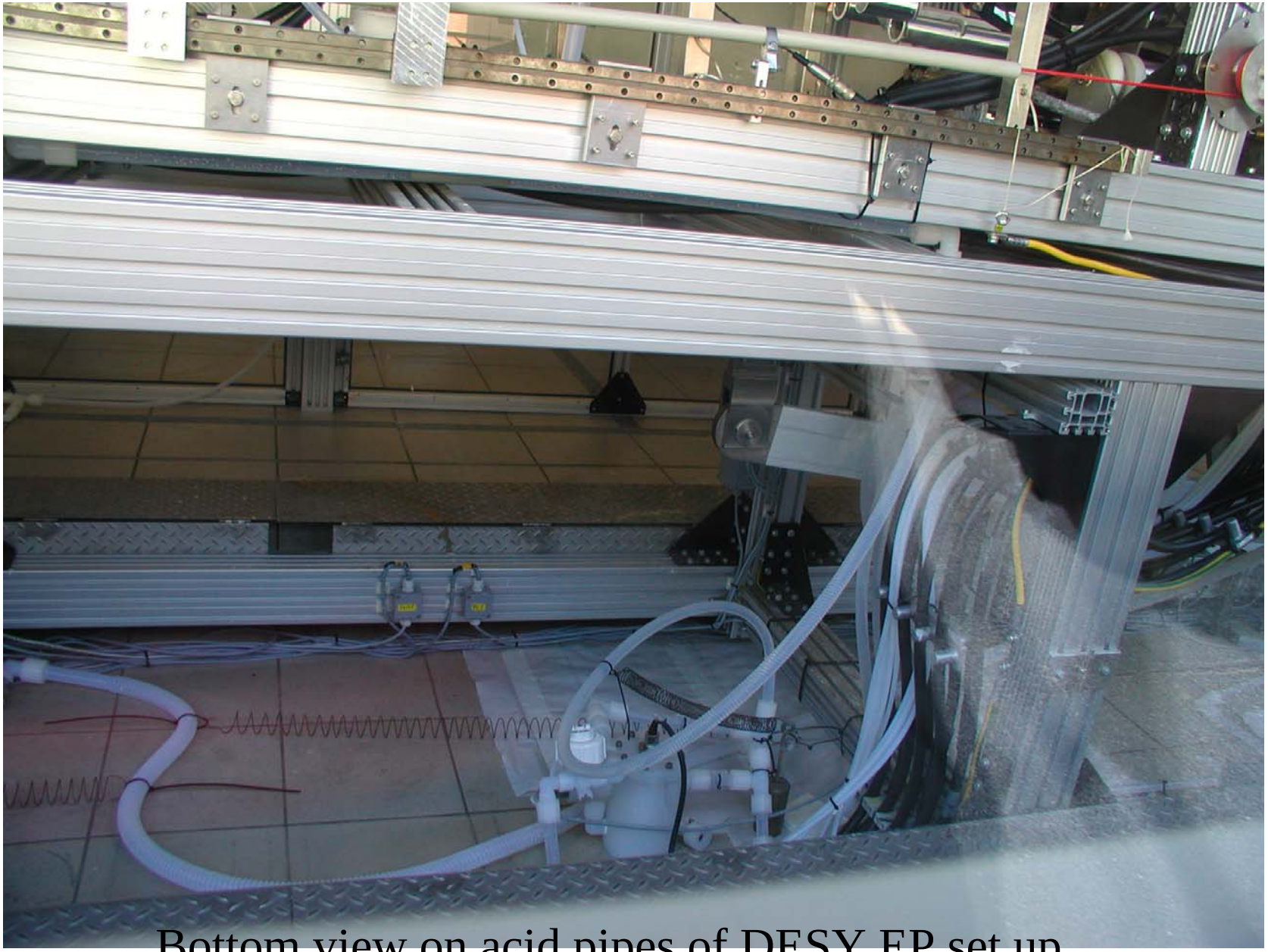
- Dump of rinsing water by industrial company
- Control Ph value before personal entrance the housing


INSTANT SPAN
INSTANT
WAGNER


SPAN
SPAN

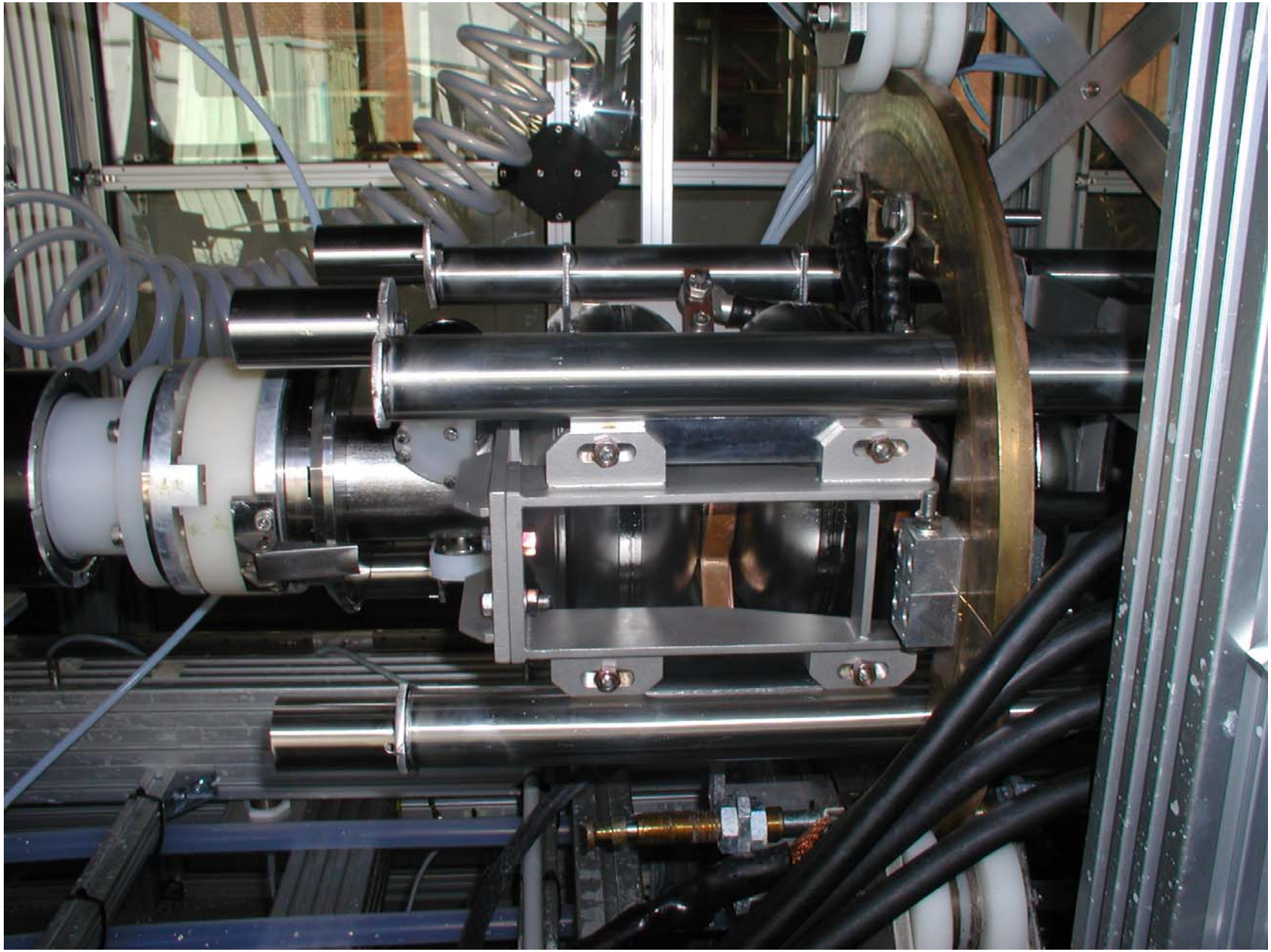

Regent
FluoroPure

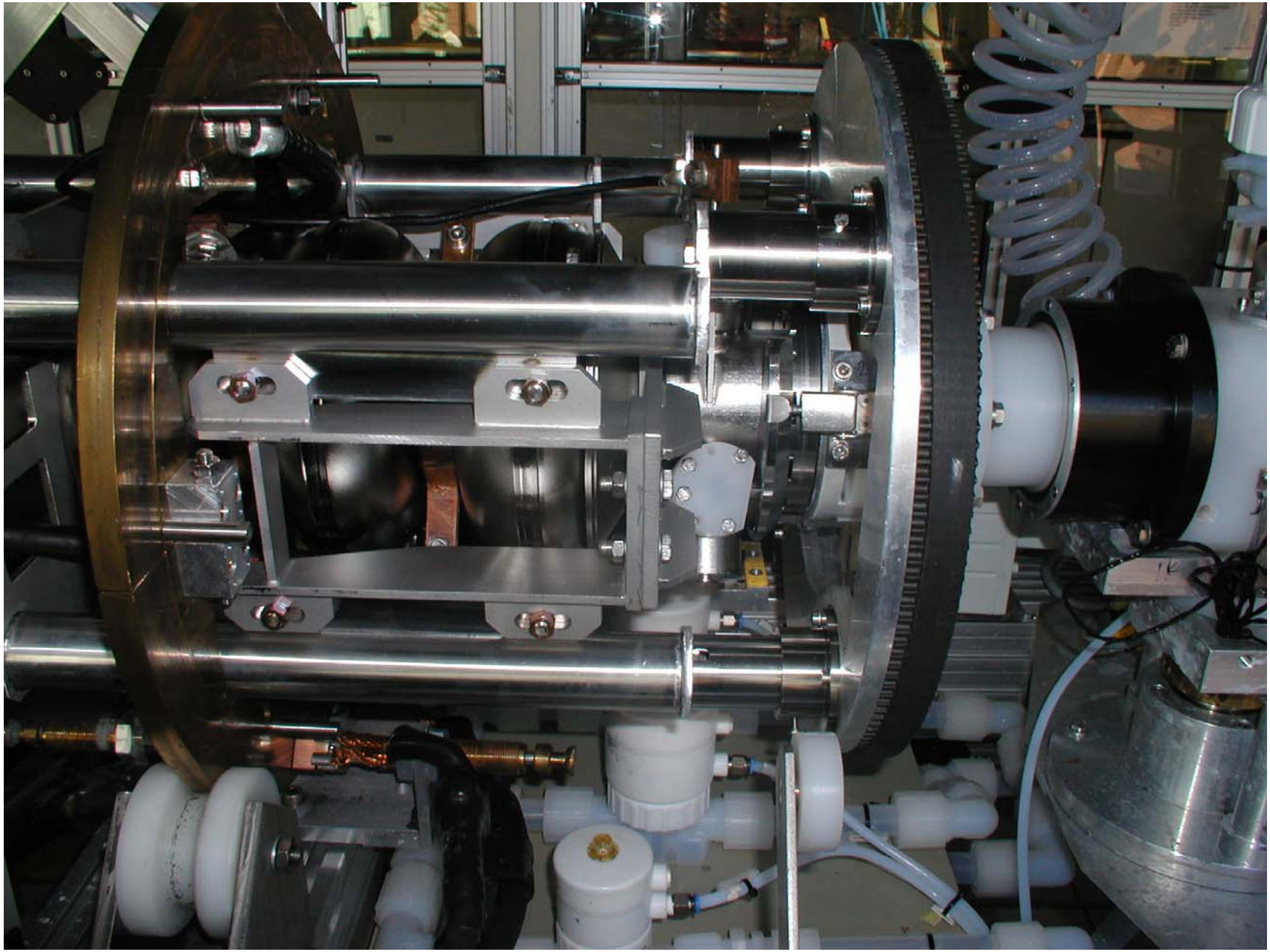

DEMAC

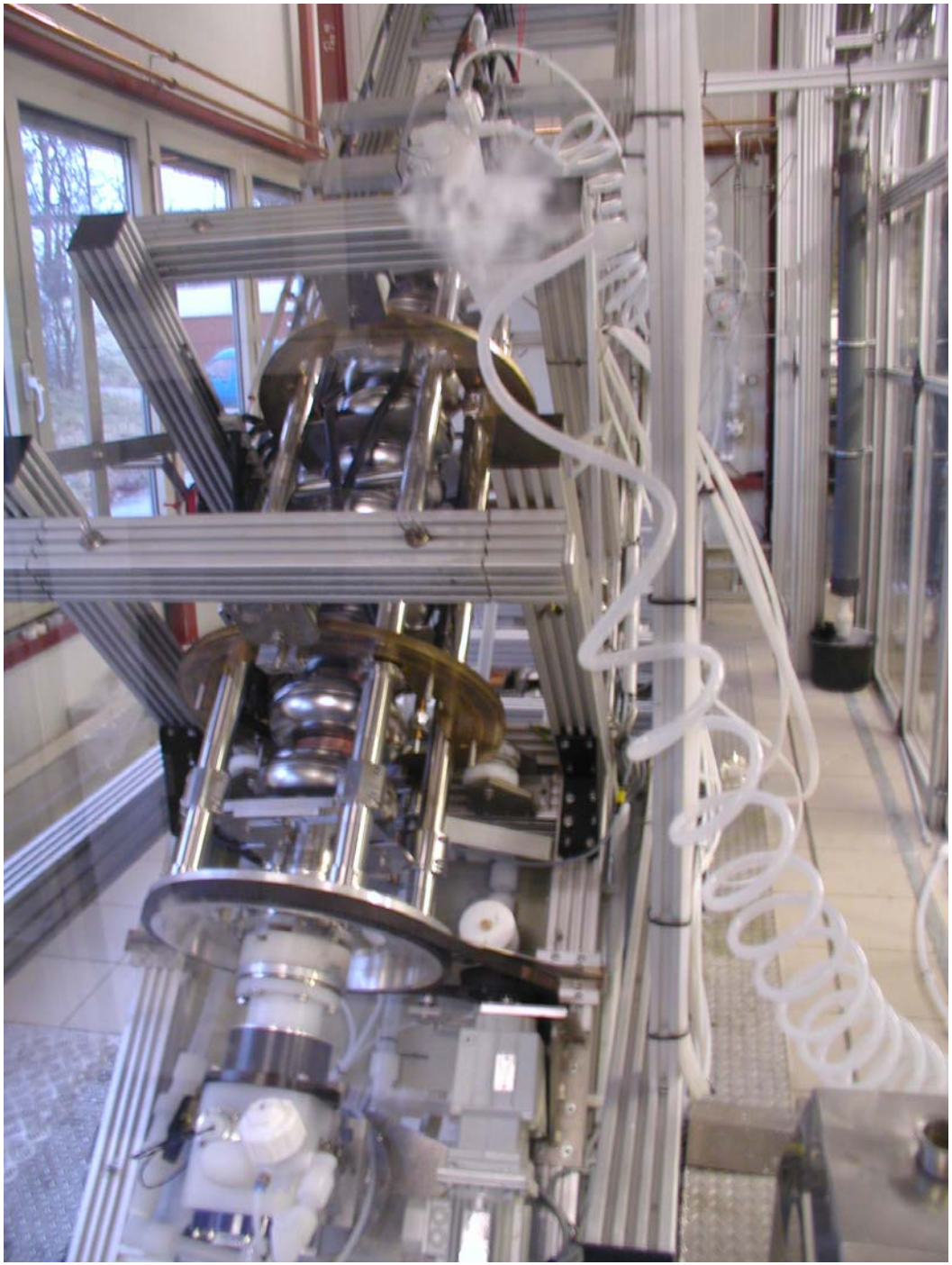


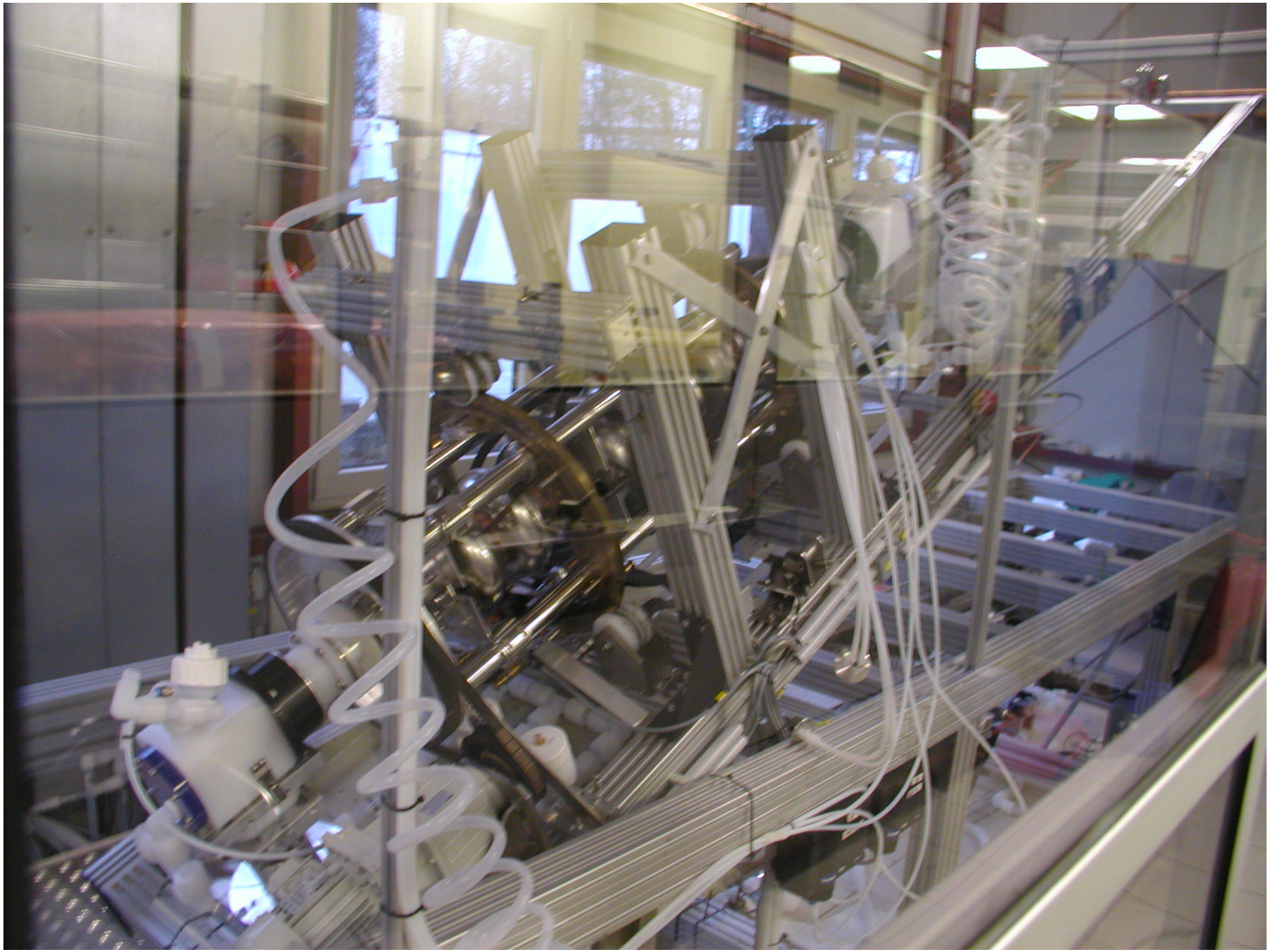
Bottom view on acid pipes of DESY EP set up

Acid sump and feed back pump of DESY EP set up









My conclusion from that for the DESIGN is :

- Contact administration before start to design/ copy
  - ➔look what you can use /apply of existing design in parallel
- Define your need before start of design /copy

  Look for space (Copy of DESY design need adaptation)
  - Different space area can give some features (not applying at DESY)
  - Basement available => Number of pumps
  - Acid volume => integrated heat exchanger (J lab)
  - Design 2*9 cell may be optimized for 9 cell only

  (application for 9 cell cavities only changes turn point of apparatus)

This reduces time afterwards and reduces redesign / Stress

After That

- Review different appraturs design and see what can be coppied

  (Jlab /DESY / KEK / Numura plating / Henkel/Cornell)

Sensors

Number is defined by law and saftey and process

HF sensors commercial sensor's

calibration 2 times Year by official service

H2 Sensors commercial sensor's

calibration 2 times Year by official service

Safety sensors

Leak

Capacitive electronic sensors

High reliability high sensitivity applied for safety

Not applicable for level control etc

Level control

read relay sensors.

Empty - stop pump 2

Full - start pump 2

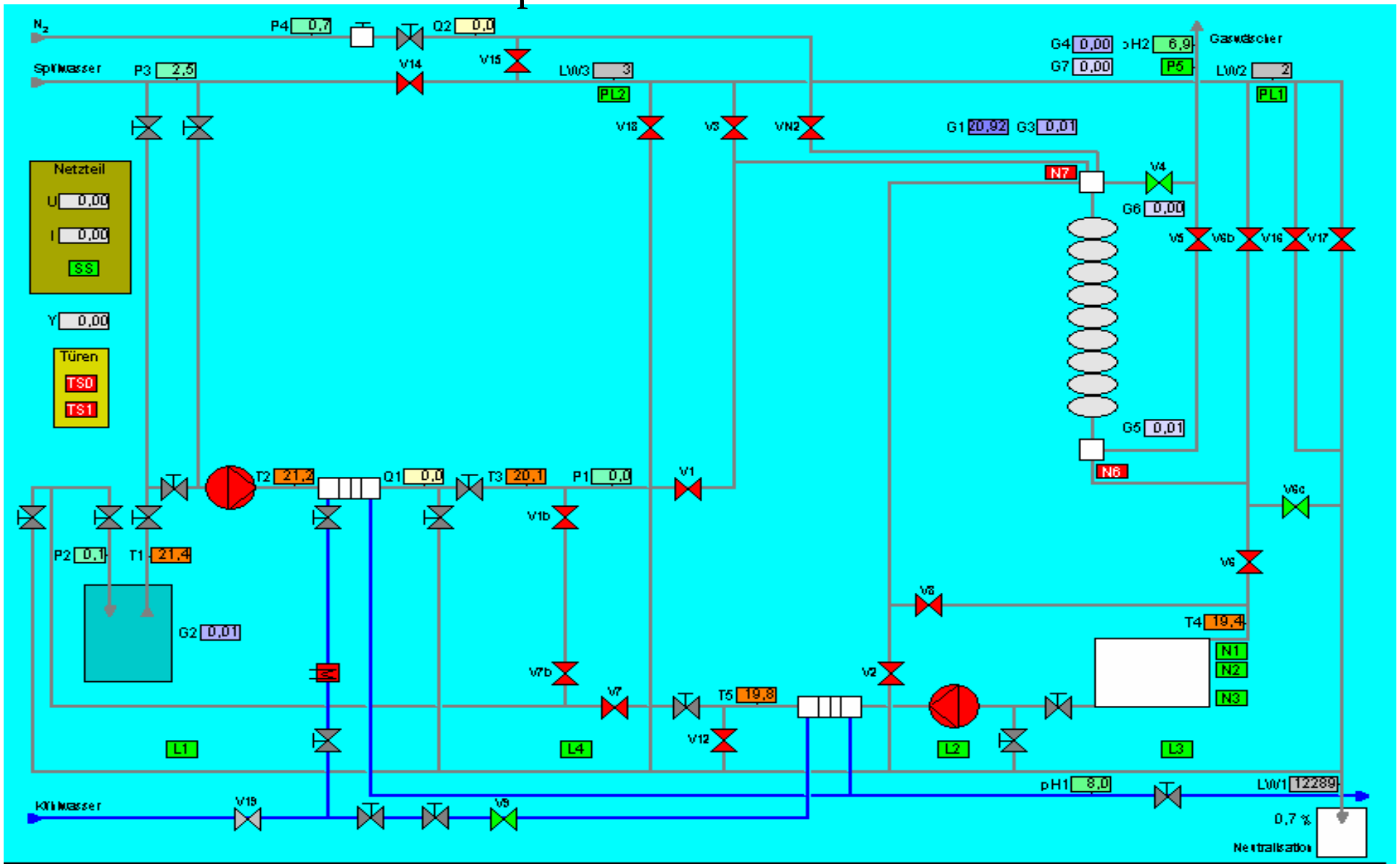
Max - stop pump 1 start pump 2

Overfilling Capacitive sensor- Stop EP

Acid Mixture 9/1 H2SO4 /HF

- process brings 6-8 KW heat to the acid for a 9 cell cavity
  - =>Temperature sensors
    - Temperature limit 35 C at 40 C (power supply off)
  - =>External single pass heat exchangers
    - designed for 20 KW for (2*9 cell)
- Leakage control
  - Pipes and acid to water connection
- Safety regulation
  - Personal needs compressed air respirators +
  - Special closings for
    - Service / maintenance / repair and exchange of barrels
- Else ? Safety department need instruction for emergency
  - coordination with fire brigade
  - training of personnel
  - extensive risk analysis for emergency and safety

Process parameters/ Sensors and control



ELSE !

Material and equipment

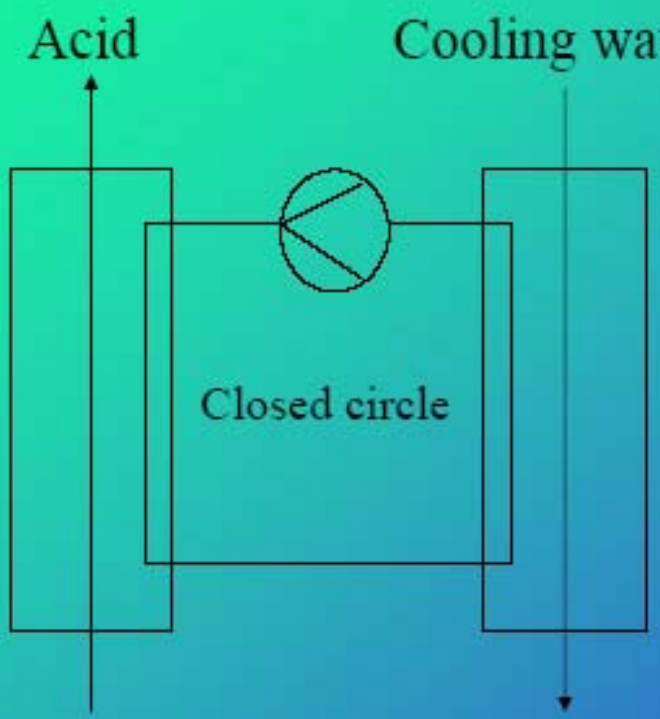
- Commercial PVDF heat exchanger failed (Leak after 3 Month)
  (about 3000 welds /Material PVDF)
- Heat exchange rate too small (Thermal conductance of H2SO4 low)
- PVDF not resistant
- Commercial HF gas scrubber failed better once too large
- Teflon heat exchanger tubes (0,8mm) wall thickness
  showed HF penetration to cooling water line
- Shaft seals made from Viton stand for 25h
- Shaft seals made from Teflon > 100h
- HF gas absorber : DESY uses granular absorbent (COSA D)

HEAT EXCHANGER

OLD VERSION

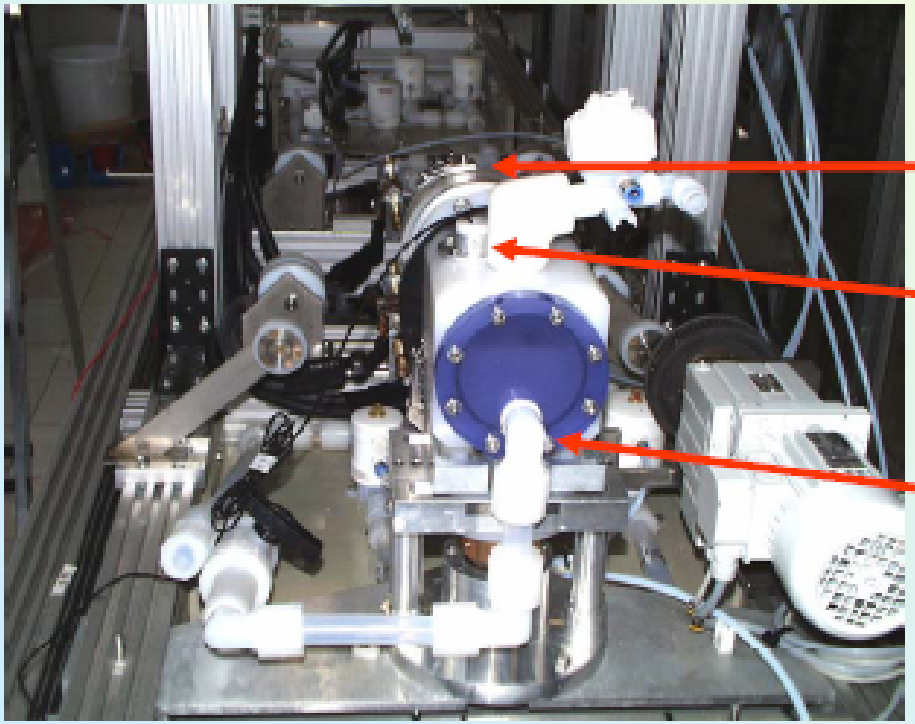
DESY VERSION

VERSION III



ELECTRO POLISHING HEADS

NEW VERSION



Solutions:

quick locking mechanism

vertical level sensors

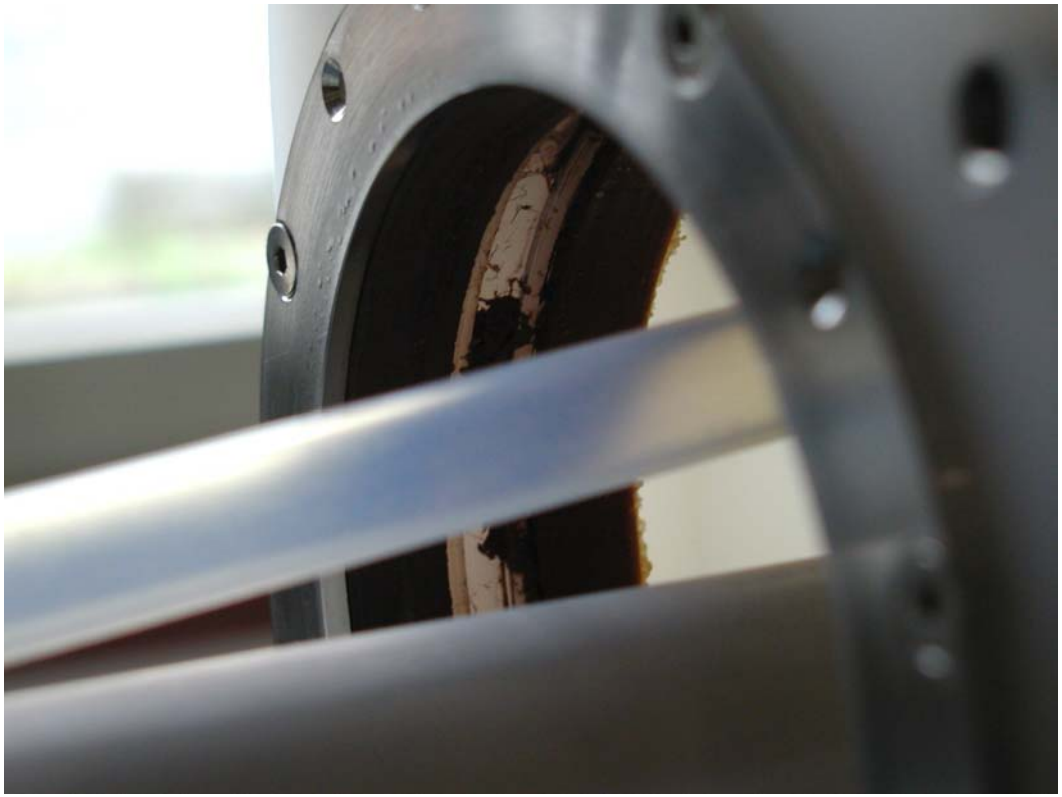
Drain now at bottom position during drain and rinse

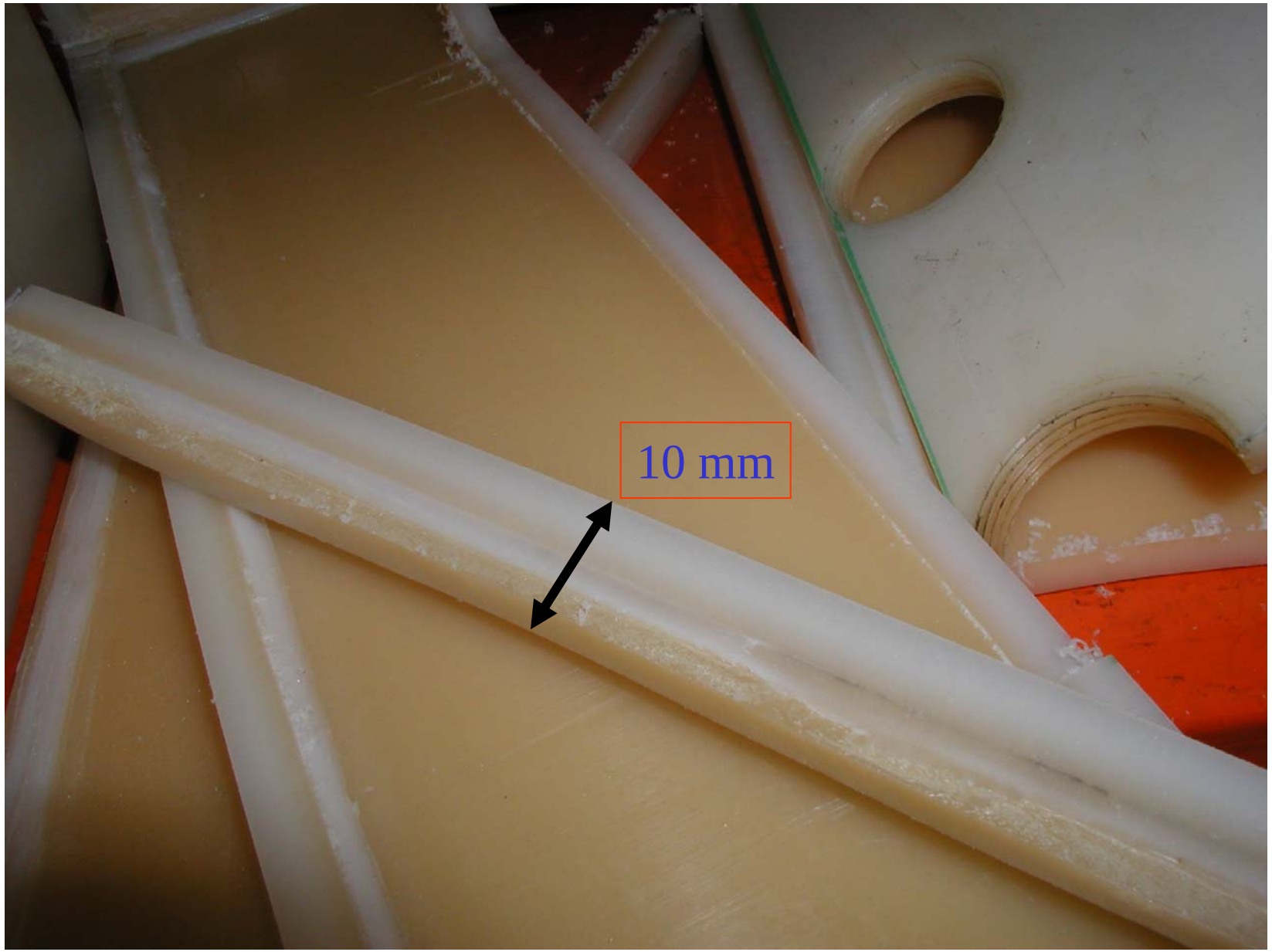
Advantage of the HF absorber opposite the gas scrubber and the lime milk cartridge

- The standard gas scrubber don´t reduce HF-gas acceptable
- The lime milk cartridge has a very good absorb rate of HF but a high danger of a blockage of the cartridge
- The pressure fluctuations in the system are reduced with the HF absorber

- The HF absorb rate of HF absorber is good
- The danger of a blockage of the absorber cartridge is clearly smaller
- Changing of the granulates is simple

Shaft seal (Viton) after 25 h of operation





PVDF tank after about 100h in operation

Process Parameters

Constant Voltage ⟷ Constant current

Optimum Voltage ?

Temperature 20C- 30C - 35C needs more investigation

Influence of T on results

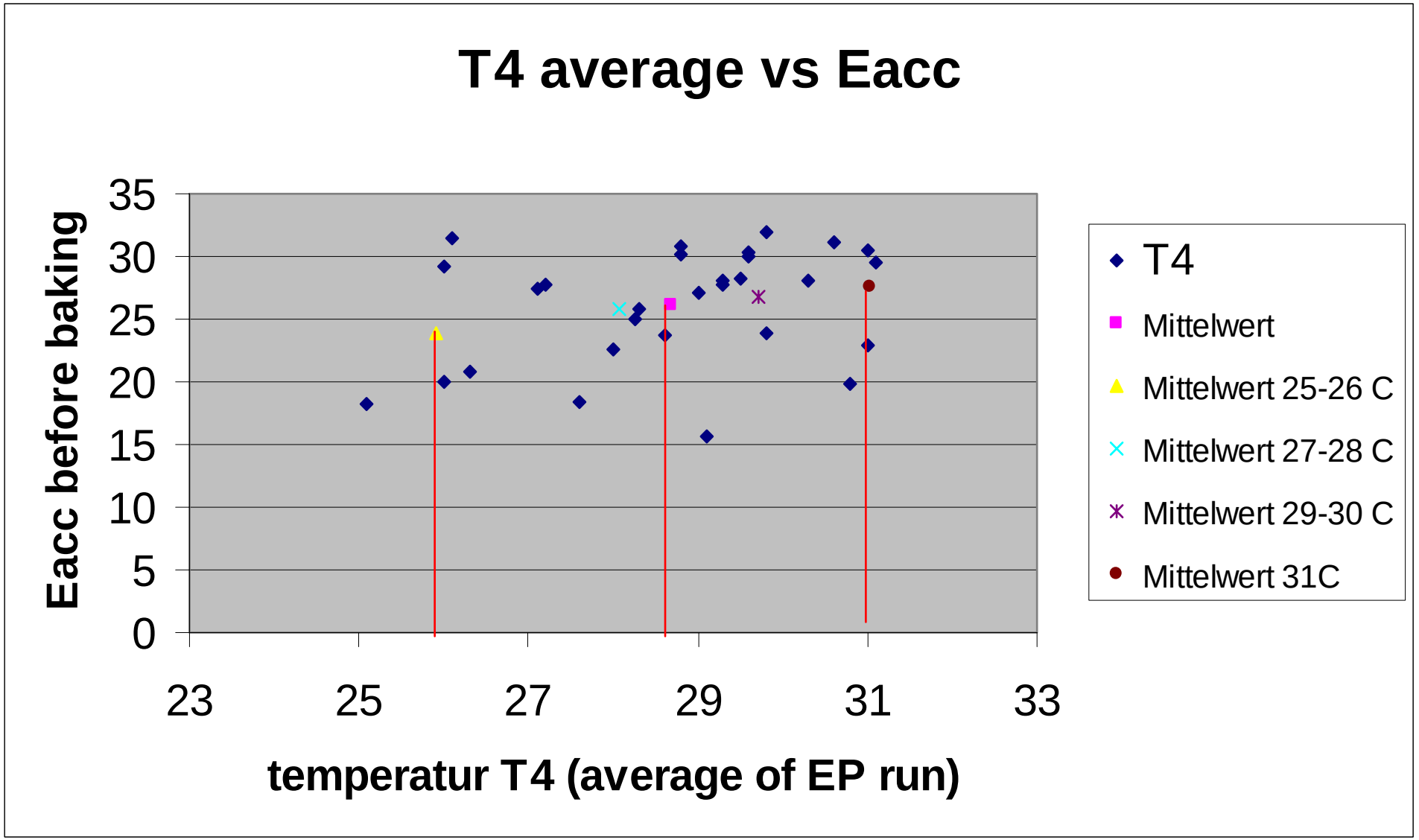
Acid HF content (Mixture)

Design of electrode

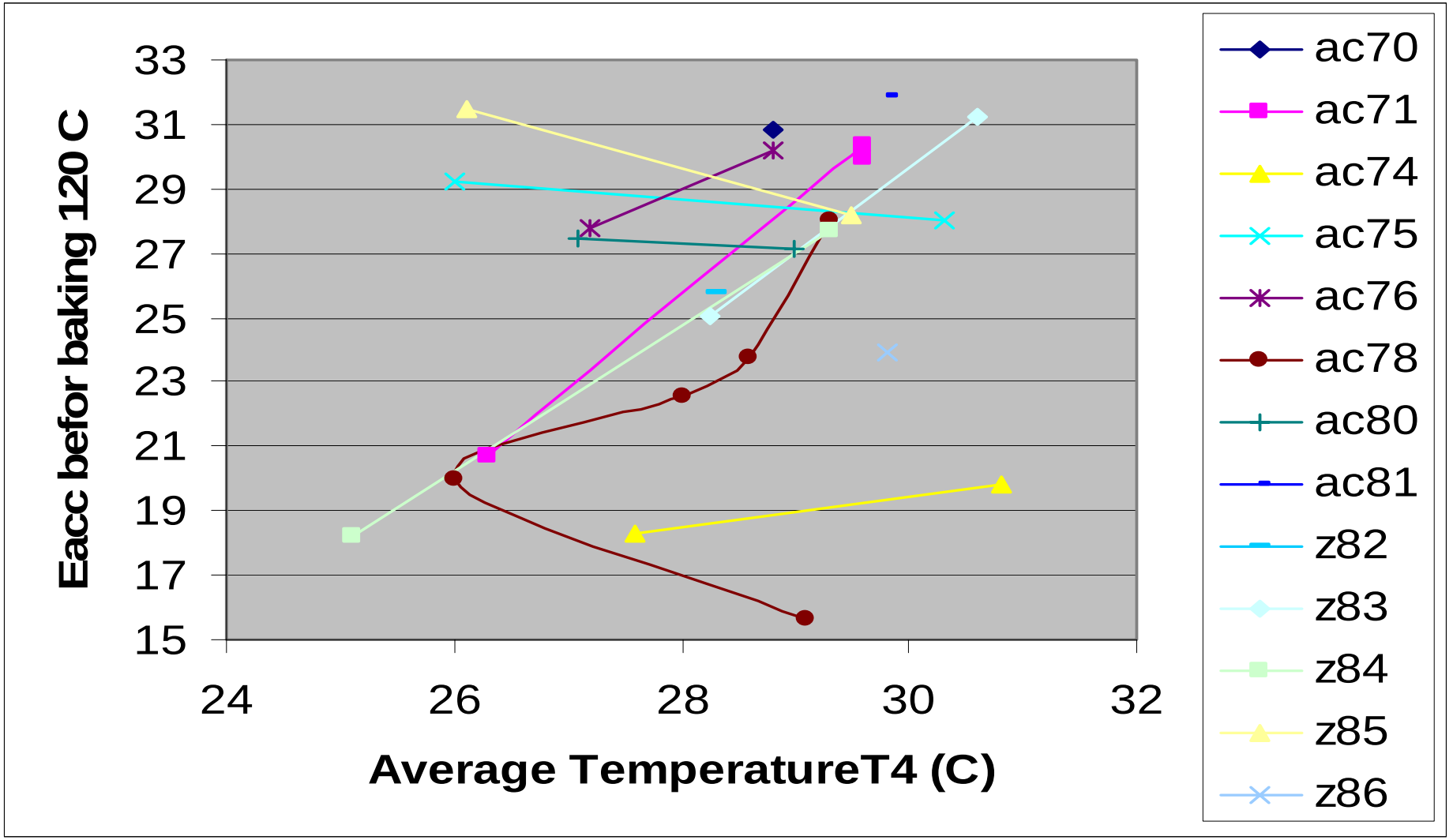
Horizontal ⟷ Vertical Polishing

Influence on Nb content in acid

	New acid (0 gr Nb/l)	Used acid (10-12 gr Nb/l)
voltage	17 [V]	17 [V]
current	320 [A]	210 [A]
Current density	5,81 [A/dm^2]	4,9 [A/dm^2]
oscillation	+44 – 66 [A]	+ 43 - 73[A]
Removal rate	0.445 [µm/min]	0,36 [µm/min]
Acid circulating	9-9,5 [l/min]	10-11 [l/min]
Acid injection speed per cell	5,3-5,6 [m/sec]	5,9-6,5 [m/sec]
Acid volume stored	150 [l]	140 [l]
HF content	2,78 [Mol/l]	~ 1,9 [Mol/l] Ref TPPT057
Nb content	0 [gr/l acid]	10-12 [gr/l acid]
Acid use time	0 [Minutes]	600 [Minutes]


T4 average vs Eacc
Eacc before baking
35
30
25
20
15
10
5
0
23
25
27
29
31
33
temperatur T4 (average of EP run)
T4
Mittelwert
Mittelwert 25-26 C
Mittelwert 27-28 C
Mittelwert 29-30 C
Mittelwert 31C

Cavity performance in respect to average Ep temperature



Parameters of the DESY Ep Set Up

•EP acid mixture: sulfuric acid (96-98 %) / Hydrofluoric acid (48 %); volume factor 9/1

•HF gases: extreme strong out gassing of HF gases during system operation

•Solubility of Nb in EP acid: 10-12 gr / liter

•**Removal rate: 0.4 µm/min (average) at V 15-18 V**

•**Current density: 9 cell cavity 5,47 A /dm2 (average)**

•Removal distribution Iris equator : iris 1,7 times higher removal than equator

(G.Kreps MHF-sl)

•**Oscillation reduction with amount on deluted niobium in the acid and reduced HF**

•Current: stronger dependency on acid volume and outlet temperature than on gradient In/out

typical values of 9 Amps /deg.**C**

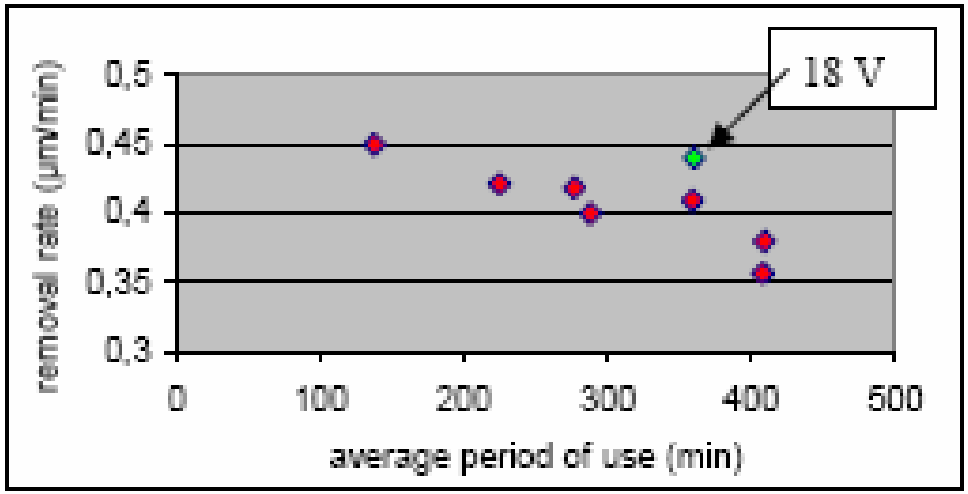
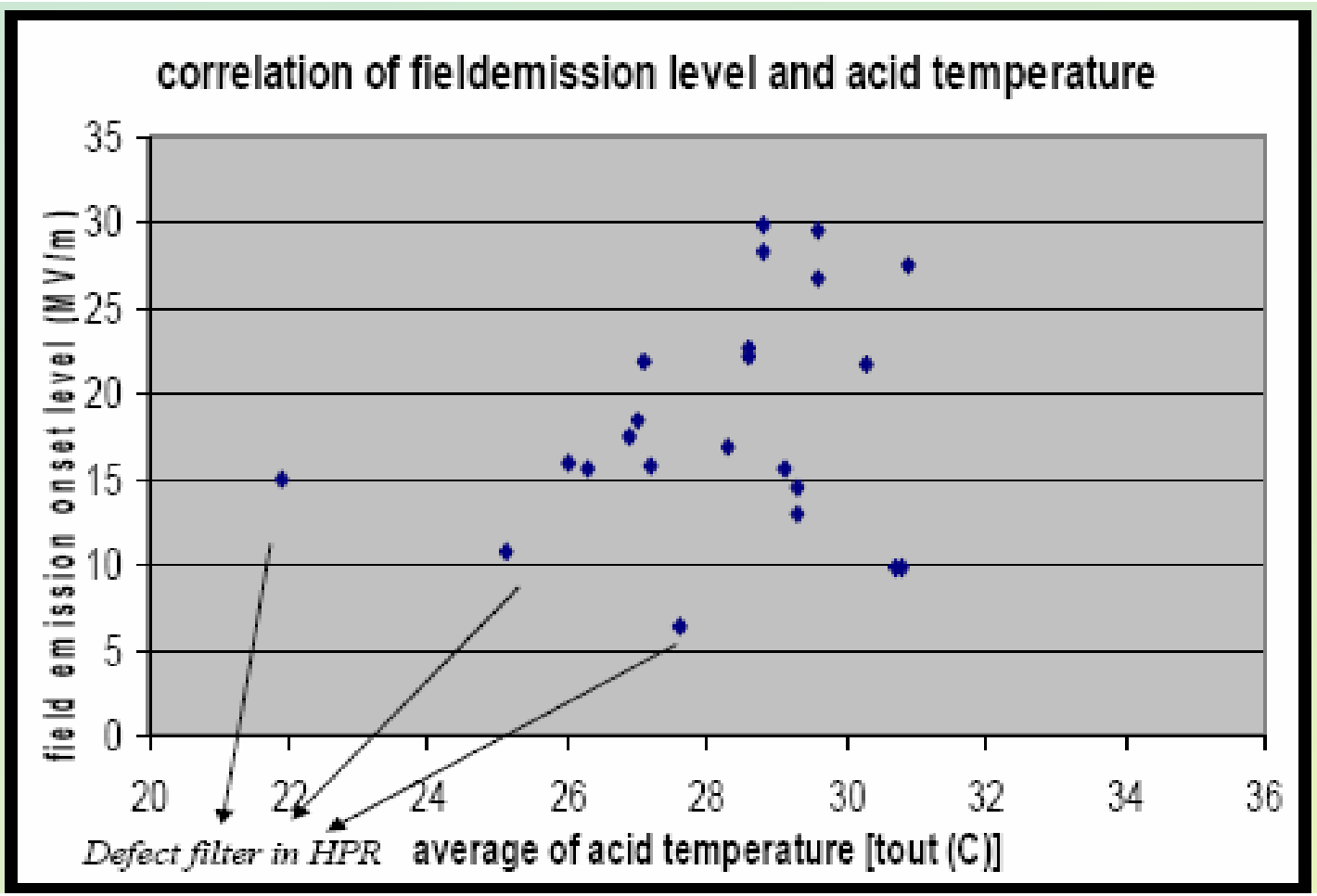
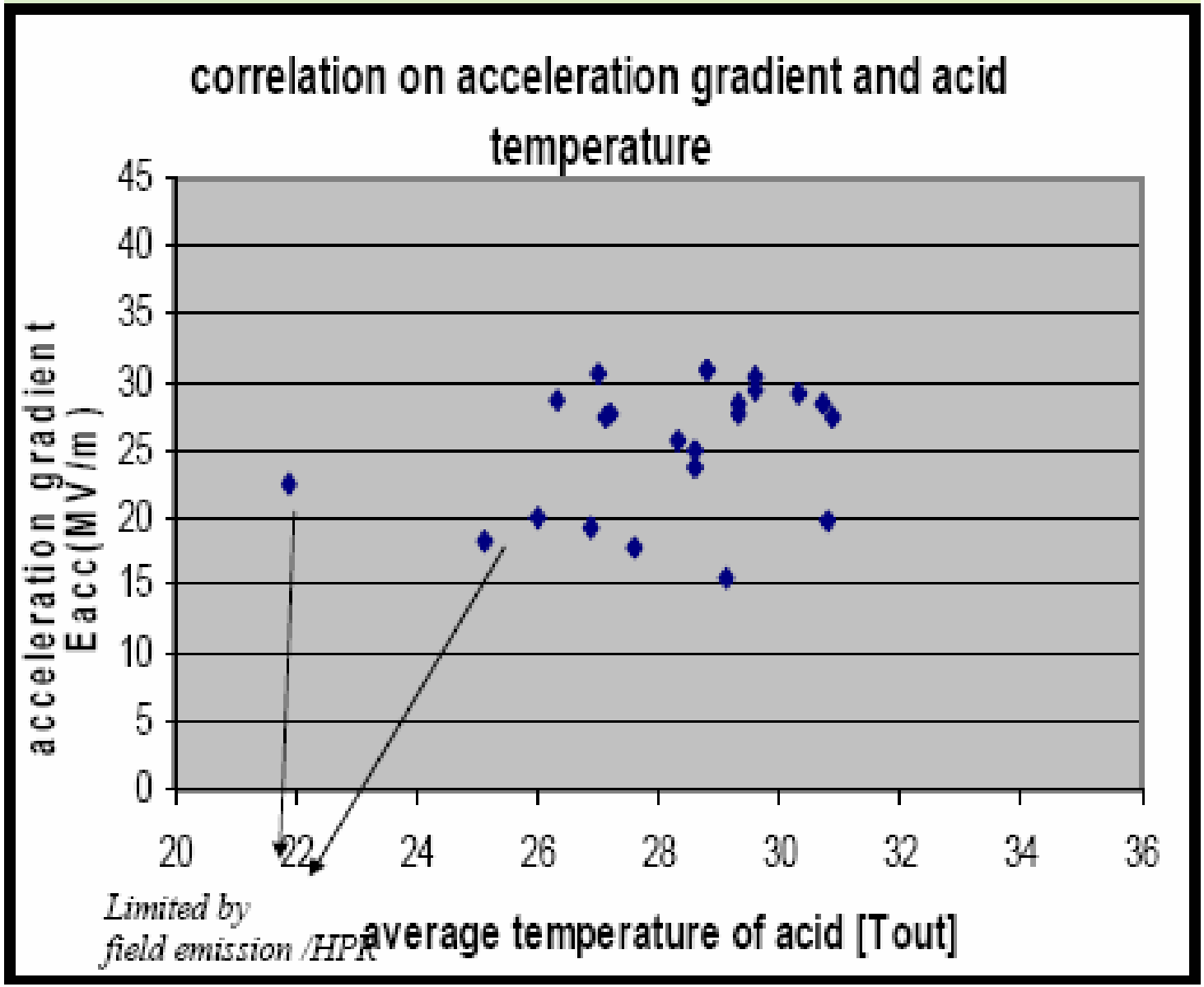


Figure 4: Removal rates and aging of the EP Mixture HF/H2SO4 (1/9). Removal rates measured for U=17V constant voltage.


correlation of fieldemission level and acid temperature
field emission onset level (MV/m)
35
30
25
20
15
10
5
0
20
22
24
26
28
30
32
34
36
Defect filter in HPR
average of acid temperature [tout (C)]


correlation on acceleration gradient and acid temperature
acceleration gradient Eacc(MV/m)
45
40
35
30
25
20
15
10
5
0
20
22
24
26
28
30
32
34
36
Limited by field emission /HPR
average temperature of acid [Tout]

DESY control of EP Parameters

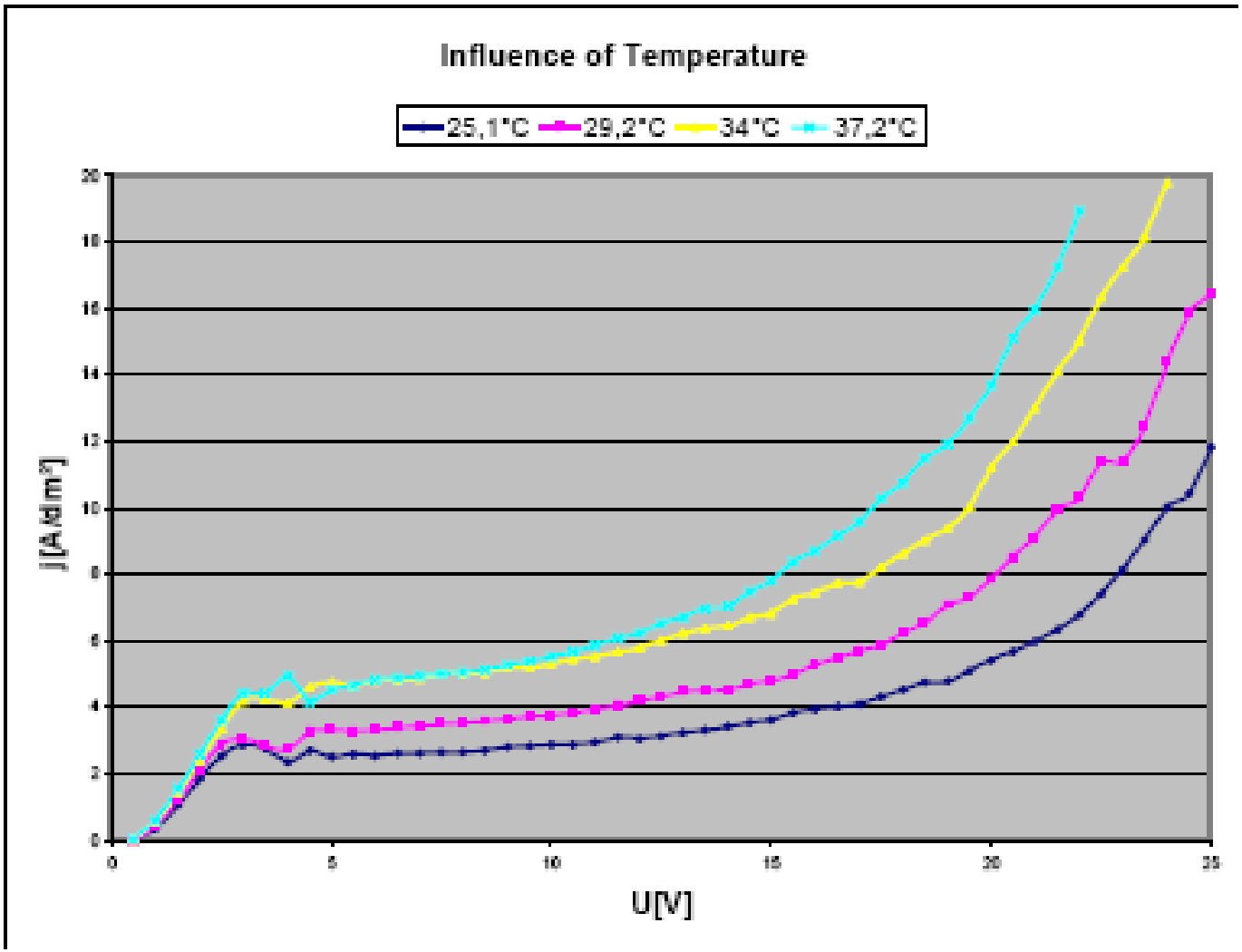
Process control by on line monitoring current and Temperature

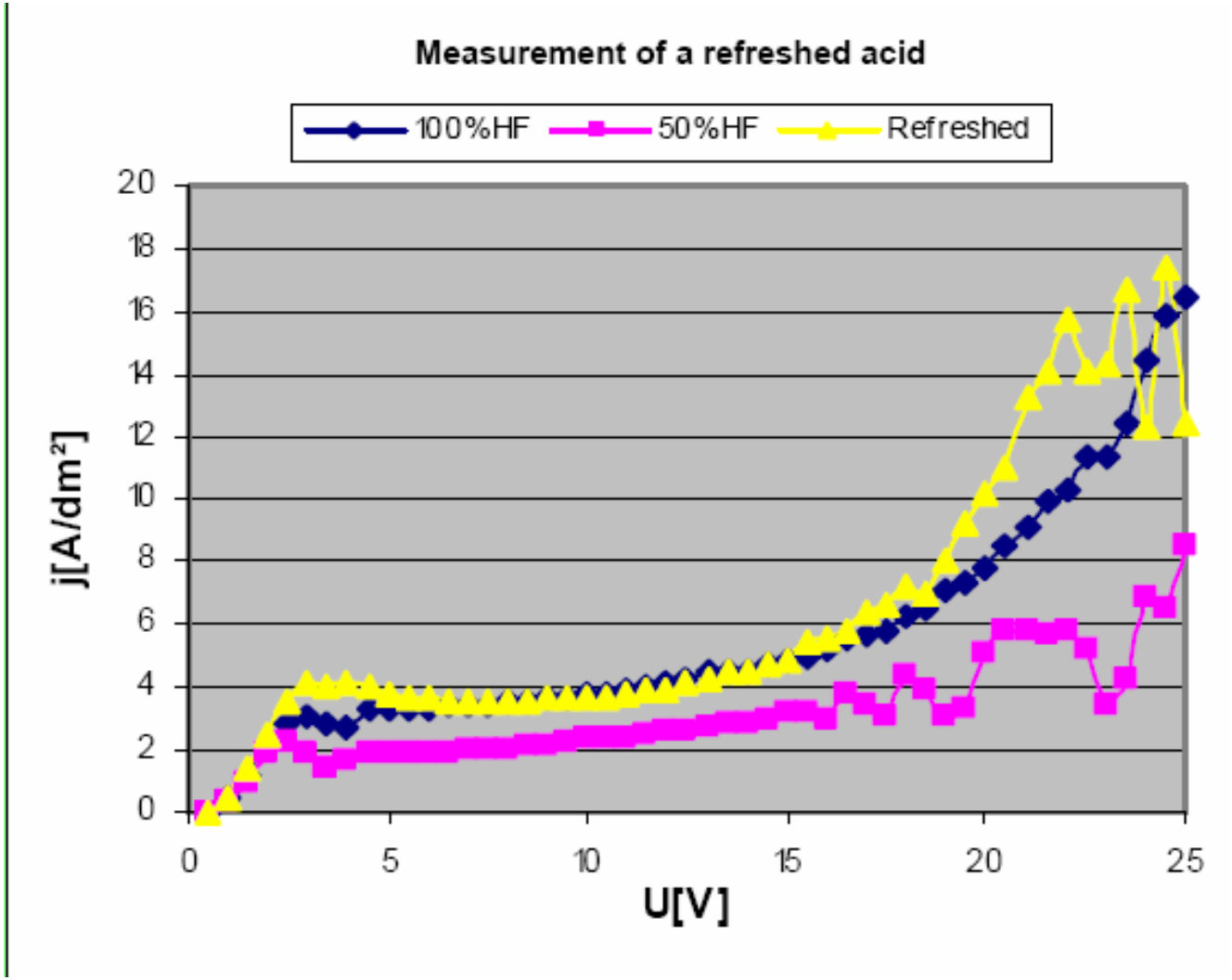
- Not stable parameters
  - Temperature
  - acid circulation (f [T])
  - HF content
  - Current (f [T;QHF])

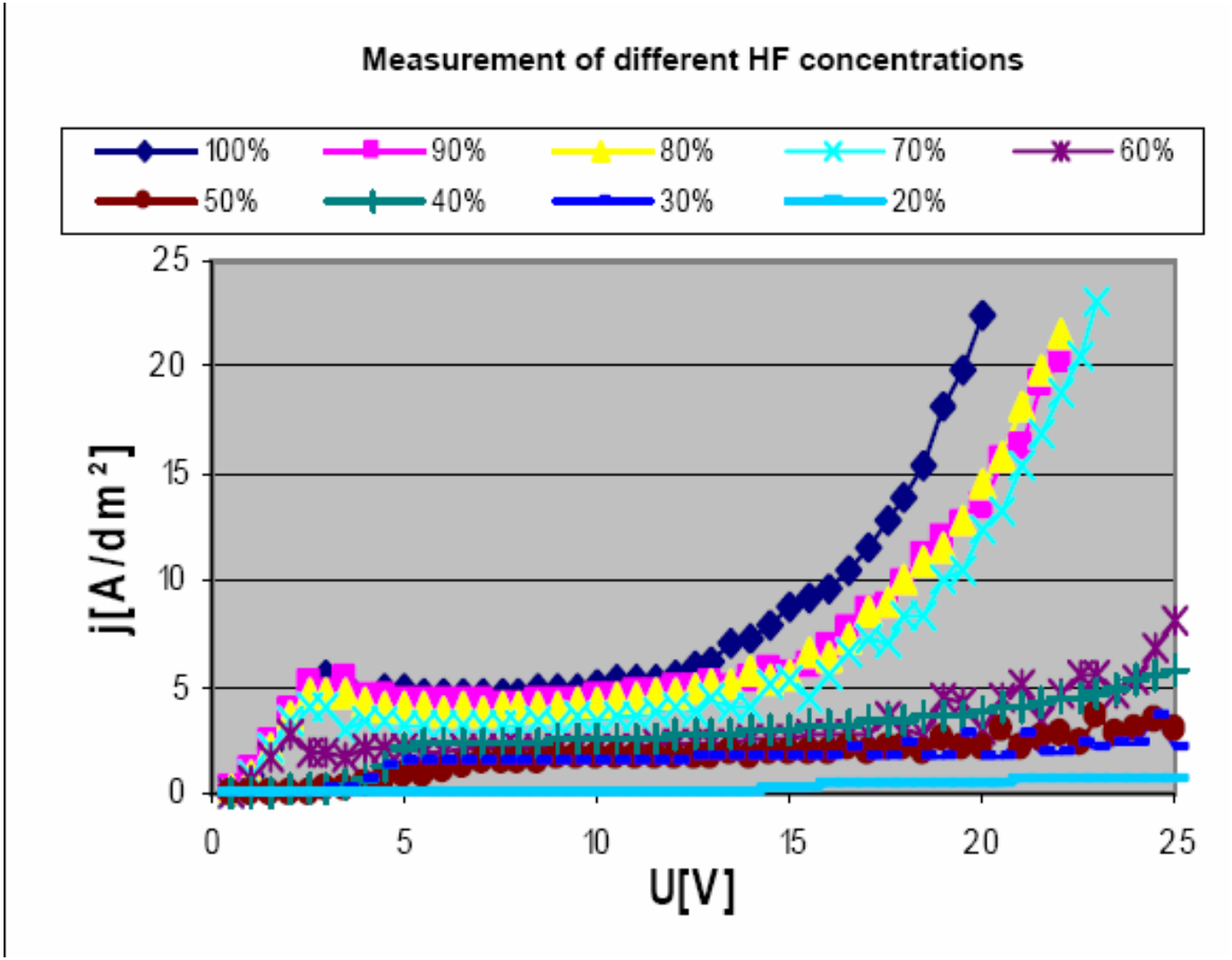
Reference measurements by Polarimetry (U/I)

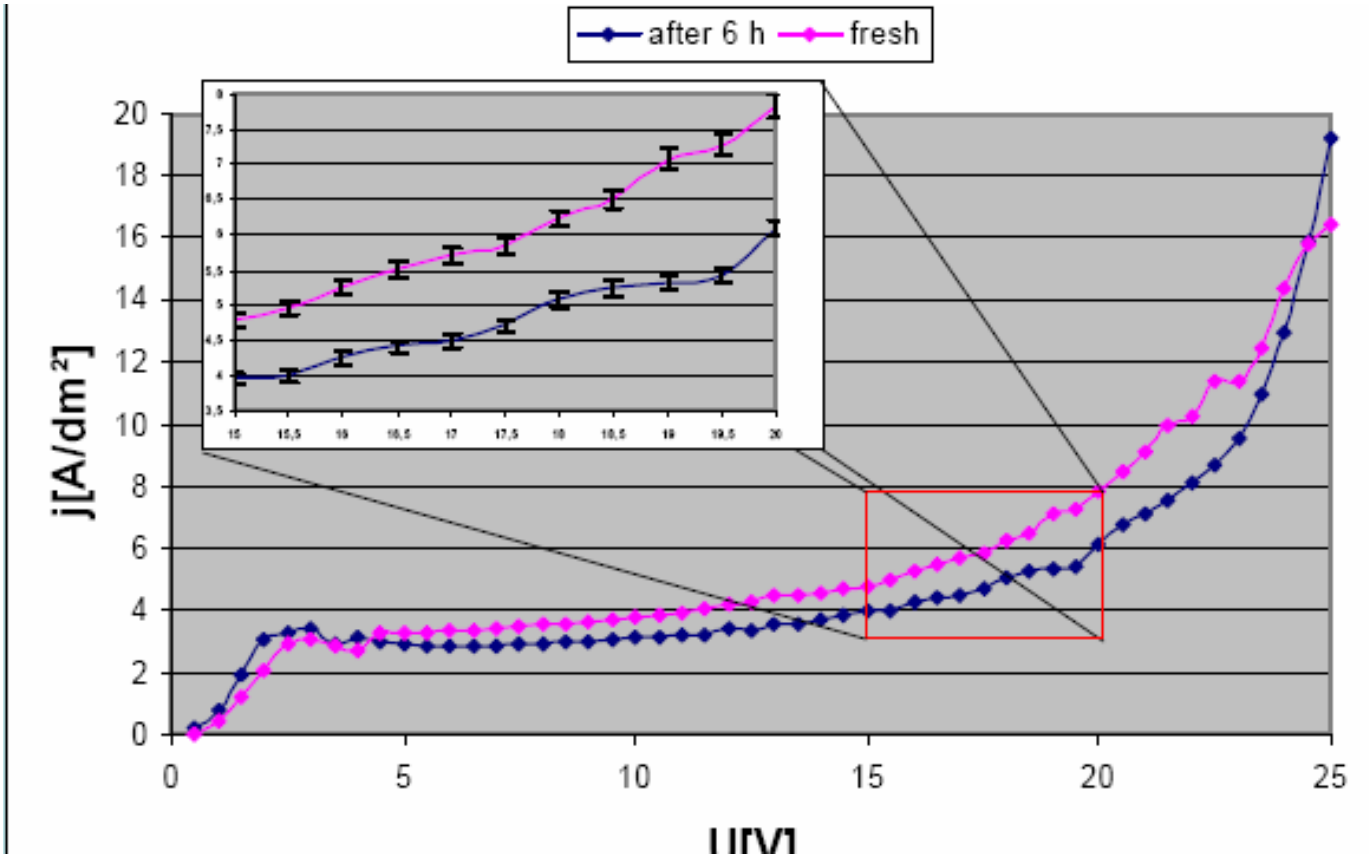
- Temperature stable
- Defined HF /Nb content

U/I Curve measured on samples /test device




Measurement of a refreshed acid
100%HF
50%HF
Refreshed
j[A/dm²]
U[V]
0
2
4
6
8
10
12
14
16
18
20
0
5
10
15
20
25


Measurement of different HF concentrations
100%
90%
80%
70%
60%
50%
40%
30%
20%
j[A/dm²]
U[V]
0
5
10
15
20
25


after 6 h
fresh
j[A/dm²]
U[V]
0
2
4
6
8
10
12
14
16
18
20
0
5
10
15
20
25

Conclusion

No clear parameter set available/ seen yet
at the DESY Infrastructure

➔ More studies on optimum parameter

DESY task =stabilize T and HF content

Reduce number of variables to get parameter

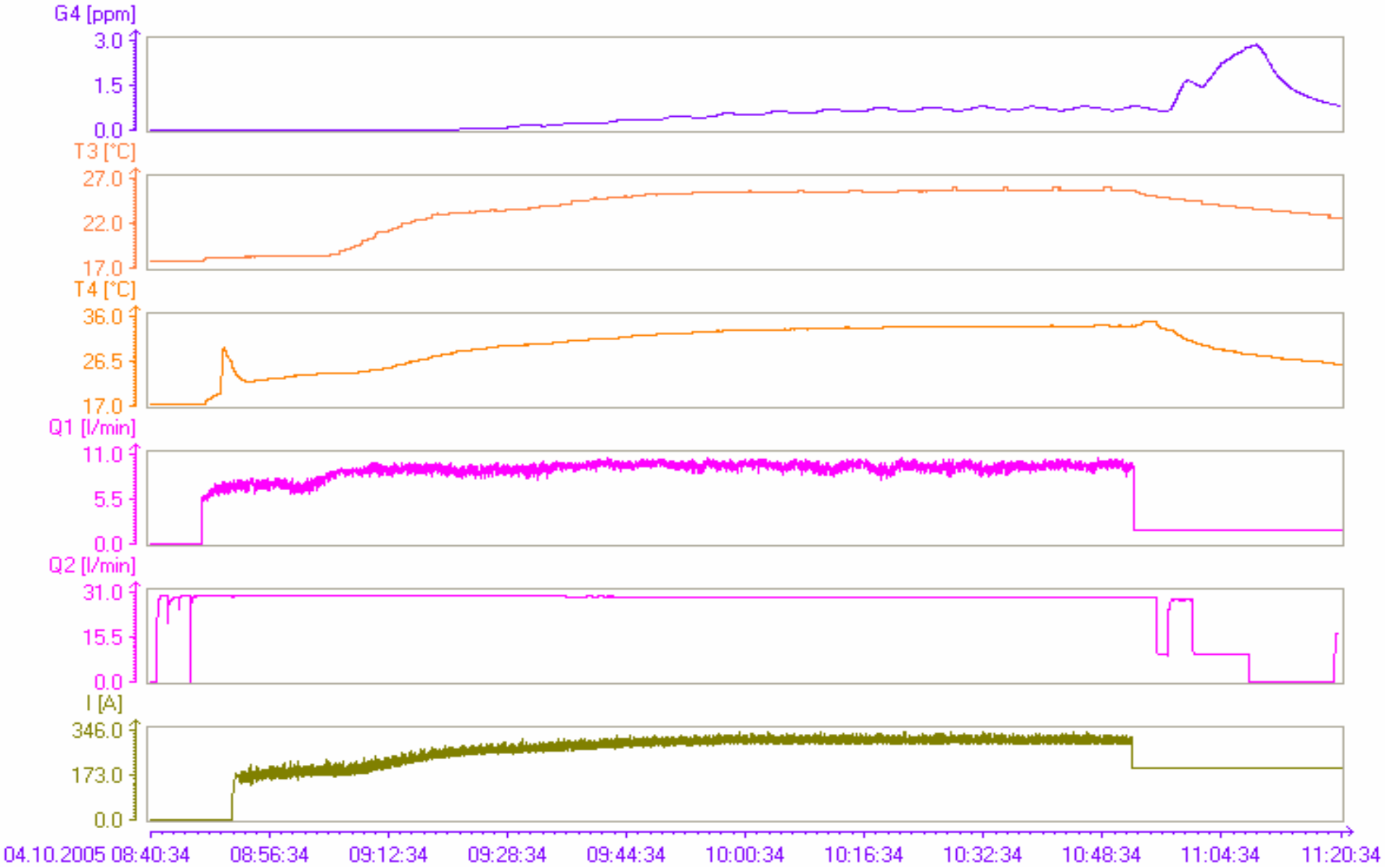
- Temperature
- Mixture/ HF content

to be tested on single cells ???

- Sulfur sedimentation
- actually unknown is influence of ??

More online process parameters need to be observed

➔Install Integrated U-I sampler from the beginning
(see Jlab)



Monitoring the EP run of a 9 cell cavity

Acid mixing / transport and storage

We learned that acid mixture is not acid mixture !!

Actually first results on “COMPOSITION”

H_2S + H_2SO_4 +HF <-> FSO_3H +H_2O +HF +X Nb?

H_2S + H_2 SO4 +HF <-> SO_2F_2+H_2O +HF(>0.1%) +X Nb??

- But Cavity result looked good (candidate for 35 MV/m)
- But U- I measurements look fine
- But standard analysis “titration of acid “ looked fine
- Apparatus stopped because HF exhausting >>>3ppm after 35 min

We expect written down information by end of October

My Summery of this is

- We need to have acid quality management
- We need to define the instruments for analysis of EP
- I would like to understand what the chemical reactions are that we use for EP (a new parameter ???)

From that it may become clearer which parameters are the once needed for high gradient

Exchange of knowledge

to be continued